

## **REMARKS**

Further and favorable reconsideration is respectfully requested in view of the foregoing amendments and following remarks.

### **Claim Amendments**

Claim 25 has been amended to limit the polyamide (co-)polymer to polymetaxylylene adipamide, and to clarify that the water-soluble compound is an inorganic electrolyte. Support for these amendments is found on page 14, lines 17-18 of Applicants' specification. As a result of the amendments to claim 25, claims 34 and 35 have been cancelled, without prejudice. No new matter has been added to the application by these amendments.

### **Rejection Under 35 U.S.C. § 112, First Paragraph**

The rejection of claim 35 under 35 U.S.C. § 112, first paragraph, as failing to comply with the written description requirement has been obviated by the cancellation of this claim.

### **Rejection Under 35 U.S.C. § 112, Second Paragraph**

The rejection of claims 25 and 35 as being indefinite under 35 U.S.C. § 112, second paragraph has been obviated by the amendment to claim 25 and the cancellation of claim 35.

Specifically, the Examiner states that it is not clear as to whether the water-soluble compound comprises an inorganic electrolyte or whether the hot water contains an inorganic electrolyte. However, it is clear from amended claim 25 that the hot water contains a water-soluble compound, which is an inorganic electrolyte.

### **Prior Art Rejections**

The patentability of the present invention over the disclosures of the references relied upon by the Examiner in rejecting the claims will be apparent upon consideration of the following remarks.

**Rejection Under 35 U.S.C. § 102 and 35 U.S.C. § 103 Based on Tanaka et al.**

Thus, the rejection of claims 25-28, 31, 34 and 39-41 under 35 U.S.C. § 102(b) as being anticipated by Tanaka et al., as well as the rejection of claims 28, 29, 32 and 33 under 35 U.S.C. § 103(a) as being unpatentable over Tanaka et al. are respectfully traversed.

The Examiner takes the position that Tanaka et al. disclose a heat treating method for a packaging product, comprising providing a packaging product formed by enclosing a content material within a packaging material comprising at least a layer of hydrophilic gas-barrier resin selected from the group consisting of ethylene-vinyl alcohol copolymer, polyamide copolymers and glycolic acid copolymers. The Examiner also states that Tanaka et al. disclose using polyvinyl alcohol and polymers of polyamide. The Examiner further states that Tanaka et al. disclose a water soluble compound comprising an inorganic electrolyte being added to the water.

Regarding claims 28 and 29, the Examiner admits that Tanaka et al. are silent in specifically disclosing wherein the hot water contains the water-soluble compound at a concentration exceeding 0.1 wt% and further wherein the hot water contains the water-soluble compound at a concentration of at least 1 wt.%. The Examiner asserts, however, that these limitations would have been obvious.

Regarding claims 32 and 33, the Examiner also admits that Tanaka et al. do not specifically disclose wherein the water-soluble compound is selected from the group consisting of sodium chloride, magnesium chloride and potassium chloride. However, the Examiner asserts that this limitation does not provide a patentable feature over the prior art.

The process of Tanaka et al. is directed to the production of a gas barrier film having a gas-barrier layer formed by crosslinking poly(meth)acrylic acid (A) with polyalcoholic polymer (B), represented by polyvinyl alcohol, and with a polyvalent metal (C). The method of the present invention is a heat-treating method for a packaging product by heat-treating the packaging product with hot water containing a water-soluble inorganic electrolyte, wherein the packaging material comprises at least a layer of a specific hydrophilic gas-barrier resin selected from the group consisting of ethylene-vinyl alcohol copolymer, polymetaxylylene adipamide and glycolic acid (co-)polymer, each of which exhibits a gas-barrier property by itself. Thus, the main distinction between the present invention and Tanaka et al. is the failure of Tanaka et al. to teach or suggest the use of a specific hydrophilic gas-barrier resin

selected from the group consisting of ethylene-vinyl alcohol copolymer, polymetaxylylene adipamide and glycolic acid(co-)polymer.

Additionally, the present invention prevents opalescence of a packaging material comprising a hydrophilic gas-barrier resin selected from the group consisting of ethylene-vinyl alcohol copolymer, polymetaxylylene adipamide and glycolic acid copolymer during hot water treatment thereof by causing the hot water to contain an inorganic electrolyte (as represented by sodium chloride) as a water soluble compound. As discussed in the prior response, Applicants' invention is based on a concept that lowering the percentage of free water molecules (by adding a water-soluble compound in hot water to cause hydration thereof with the water molecules) will decrease the kinetic energy of the water molecules, thus suppressing the opalescence of the packaging material including a hydrophilic resin layer. (See page 5, line 27 to page 6, line 11 of Applicants' specification.)

The Examiner cites column 3, lines 21-22 and column 6, lines 54-59 of Tanaka et al. as disclosing the use of polyvinyl alcohol. However, this is different from the ethylene-vinyl alcohol copolymer recited in Applicants' claims because polyvinyl alcohol is not a gas-barrier resin. Enclosed herewith, as Attachment A, is an excerpt from McGraw-Hill Dictionary of Scientific and Technical Terms, which identifies polyvinyl alcohol as a water soluble polymer. Thus, polyvinyl alcohol is not usable as a gas-barrier material, by itself, and cannot be compared to ethylene-vinyl alcohol copolymer. Enclosed herewith, as Attachment B, is an article which frequently discusses ethylene-vinyl alcohol copolymer as a gas-barrier material.

Applicants' independent claim 25 specifically recites, "at least a layer of hydrophilic gas-barrier resin selected from the group consisting of ...". Polyvinyl alcohol, as taught by Tanaka et al., is only meaningful when present together with poly(meth)acrylic acid to form a crosslinked structure. Therefore, the reference fails to teach or suggest a layer of hydrophilic gas-barrier resin selected from the group consisting of those recited in Applicants' amended claim 25. The ethylene-vinyl alcohol copolymer, polymetaxylylene adipamide and glycolic acid (co-)polymer recited in Applicants' claims are hydrophilic gas-barrier resins by themselves, i.e., without crosslinking.

As discussed above, claim 25 has been amended to limit the polyamide copolymer to polymetaxylylene adipamide, which exhibits the best gas-barrier property among the

polyamide copolymers. The reference fails to teach or suggest polymetaxylylene adipamide, as recited in amended claim 25. Polyamides, as disclosed in column 8, lines 28-30 of the reference, are not generally categorized as gas-barrier resins, in view of the very inferior gas-barrier property when compared to polymetaxylylene adipamide. Polymetaxylylene adipamide (MXD6), discussed on page 14, lines 17-18 of Applicants' specification, is a special polyamide and has superior gas-barrier property compared to other polyamines (Nylon 6 and Novamid X21), i.e., a lower oxygen permeability at a ratio of one third to one tenth. Please see Fig. 2 on page 79 of Attachment B.

Further, polyvinylidene fluoride, as disclosed in column 8, lines 34-35 of the reference, is a gas-barrier resin, but not a hydrophilic resin, as required by Applicants' claims. Polyvinylidene chloride (PVDC), as disclosed in column 8, lines 38-38 of Tanaka et al., is a well known hydrophobic gas-barrier resin. Please see claim 4 of Attachment C (U.S. '907), and claim 4 in combination with claim 1 of Attachment D (U.S. '534). The gas-barrier property not changing with a change in humidity, as shown in Fig. 1 of Attachment B is another indication of the hydrophobic nature of PVDC.

Thus, Tanaka et al. fail to teach or suggest a hydrophilic gas-barrier resin selected from the group consisting of ethylene-vinyl alcohol copolymer, polymetaxylylene adipamide and glycolic acid (co-)polymer, as required by Applicants' claims.

Tanaka et al. disclose, as a second embodiment, a retort method characterized by treating a container for retort packaging comprising a laminated film comprising a layer formed of a cross-linked structure containing ester bonds between poly(meth)acrylic acid (A) and a polyalcoholic polymer (B) in water containing a metal (C). The treatment is performed for forming a new ionic crosslinked structure. (See column 3, lines 21-25 and 41-51 of the reference.) More specifically, the ions of the metal (C) permeate into the film to form ionic cross-linking with free carboxylic acids originating from poly(meth)acrylic acid. (See column 11, lines 40-54 and chemical structure (X) in column 4 of the reference.) As the metal (C), polyvalent metals are used. (See column 11, lines 6-8 of the reference.)

Therefore, Tanaka et al. disclose the formation of a gas-barrier layer comprising a poly(meth)acrylic acid crosslinked with polyvalent metal ions. This type of gas-barrier layer is quite different from that recited in Applicants' claims, i.e., a hydrophilic gas-barrier layer selected from the group consisting of ethylene-vinyl alcohol copolymer, polymetaxylylene

adipamide and glycolic acid (co-)polymer. Applicants' recited gas-barrier layer is free from a cross-linked structure and also essentially free from free acid groups required by crosslinking with polyvalent metal ions. Although the claim does not specifically recite these limitations, it is implicit due to the non-crosslinked nature arising from the specific resin recited in claim 25. Specifically, glycolic acid is a hydroxy carboxylic acid and glycolic acid (co-)polymer is a completely esterified polymer substantially free from free acid groups, in contrast to the poly(meth)acrylic acid used in Tanaka et al.

Additionally, in Applicants' invention, an inorganic electrolyte, as represented by sodium chloride (claim 33) having a high water-solubility and lacking a crosslinked structure-forming ability, is preferred, and a polyvalent metal salt having a low-water-solubility is not preferred.

Thus, Tanaka et al. is directed to formation of a type of gas-barrier layer which is quite different from Applicants' recited gas-barrier layer. Specifically, Tanaka et al. fail to teach or suggest the treatment of a packaging material comprising a layer of a hydrophilic gas-barrier resin selected from the group consisting of ethylene-vinyl copolymer, polmetaxylylene adipamide and glycolic acid (co-)polymer with hot water containing an inorganic electrolyte. Additionally, Tanaka et al. requires a crosslinked structure, which is clearly distinct from Applicants' recited gas-barrier layer. Finally, Tanaka et al. fail to teach or suggest the prevention of opalescence of the packaging material, as disclosed in Applicants' specification.

For these reasons, the invention of claims 25-29, 31-34 and 39-41 is clearly patentable over Tanaka et al.

**Rejection Under 35 U.S.C. § 103 Based on Tanaka et al. in View of Su**

The rejection of claim 35 under 35 U.S.C. § 103(a) as being unpatentable over Tanaka et al. in view of Su has been obviated due to the cancellation of claim 35.

**Rejection Under 35 U.S.C. § 103 Based on Tanaka et al. in View of Shiiki et al.**

The rejection of claim 42 under 35 U.S.C. § 103(a) as being unpatentable over Tanaka et al. in view of Shiiki et al. is respectfully traversed.

The Examiner admits that Tanaka et al. are silent in teaching wherein the gas-barrier resin is a glycolic acid copolymer. However, the Examiner asserts that Shiiki et al. teach a gas barrier composite film comprised of a polymer of glycolic acid for use in food packaged materials that undergo high-temperature and high humidity conditions. Further, the Examiner states that Shiiki et al. teach when said polyglycolic acid film is used in combination with a thermoplastic film that the gas barrier properties of the film are markedly improved. The Examiner states that this is analogous to the teachings of Tanaka et al. who disclose two layers wherein one layer is a gas barrier and the second layer is a thermoplastic film. The Examiner takes the position that to use a gas barrier resin comprising a polyglycolic acid copolymer would not have provided a patentable feature over the prior art, since the polyglycolic acid film would provide an equivalent function for providing a gas-barrier film.

However, Shiiki et al. fail to remedy the deficiencies of Tanaka et al., as discussed above. Specifically, even if it is known that a polymer of glycolic acid is a good gas-barrier resin, as shown in Shiiki et al., the substitution thereof for the crosslinked gas-barrier resin system of Tanaka et al. would change the intrinsic nature of the system of Tanaka et al. Combining a secondary reference which is so contrary to the teachings of a primary reference is untenable.

For these reasons, the invention of claim 42 is clearly patentable over Tanaka et al. in view of Shiiki et al.


**Conclusion**

Therefore, in view of the foregoing amendments and remarks, it is submitted that each of the grounds of rejection set forth by the Examiner has been overcome, and that the application is in condition for allowance. Such allowance is solicited.

If, after reviewing this Amendment, the Examiner feels there are any issues remaining which must be resolved before the application can be passed to issue, the Examiner is respectfully requested to contact the undersigned by telephone in order to resolve such issues.

Respectfully submitted,

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# **McGraw-Hill Dictionary of Scientific and Technical Terms**

## **Fifth Edition**

**Sybil P. Parker**  
Editor in Chief

ATTACHMENT A

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On the cover: Photomicrograph of crystals of vitamin B<sub>1</sub>.  
(Dennis Kunkel, University of Hawaii)

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In addition, material has been drawn from the following references: R. E. Huschke, *Glossary of Meteorology*, American Meteorological Society, 1959; *U.S. Air Force Glossary of Standardized Terms*, AF Manual 11-1, vol. 1, 1972; *Communications-Electronics Terminology*, AF Manual 11-1, vol. 3, 1970; W. H. Allen, ed., *Dictionary of Technical Terms for Aerospace Use*, 1st ed., National Aeronautics and Space Administration, 1965; J. M. Gilliland, *Solar-Terrestrial Physics: A Glossary of Terms and Abbreviations*, Royal Aircraft Establishment Technical Report 67158, 1967; *Glossary of Air Traffic Control Terms*, Federal Aviation Agency; *A Glossary of Range Terminology*, White Sands Missile Range, New Mexico, National Bureau of Standards, AD 467-424; *A DOD Glossary of Mapping, Charting and Geodetic Terms*, 1st ed., Department of Defense, 1967; P. W. Thrush, comp. and ed., *A Dictionary of Mining, Mineral, and Related Terms*, Bureau of Mines, 1968; *Nuclear Terms: A Glossary*, 2d ed., Atomic Energy Commission; F. Casey, ed., *Compilation of Terms in Information Sciences Technology*, Federal Council for Science and Technology, 1970; *Glossary of Stinfo Terminology*, Office of Aerospace Research, U.S. Air Force, 1963; *Naval Dictionary of Electronic, Technical, and Imperative Terms*, Bureau of Naval Personnel, 1962; *ADP Glossary*, Department of the Navy, NAVSO P-3097.

## McGRAW-HILL DICTIONARY OF SCIENTIFIC AND TECHNICAL TERMS, Fifth Edition

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resin elastomer made by the reaction of a diisocyanate to a polyester (such as the glycol-adipic acid ester); has high resistance to abrasion, oil, ozone, and high temperatures. Also known as polyester rubber. { 'pāl-ē'yūr-ə,thān 'rəb-ər }

**polyuria** [MED] The passage of copious amounts of urine. { 'pāl-ē'yūr-ə }

**polyvalent** [CHEM] Pertaining to an ion with more than one valency, such as the sulfate ion,  $\text{SO}_4^{2-}$ . Also known as multivalent; polygen. [IMMUNOL] 1. Of antigens, having many combining sites or determinants. 2. Pertaining to vaccines composed of mixtures of different organisms, and to the resulting mixed antiserum. { 'pāl-i'vā-lənt }

**polyvalent number** [COMPUT SCI] A number, consisting of several figures, used for description, wherein each figure represents one of the characteristics being described. { 'pāl-i'vā-lənt 'nəm-bər }

**polyvinyl acetal resin** See vinyl acetal resin. { 'pāl-i'vīn-əl 'as-ə,tal 'rez-ən }

**polyvinyl acetate** [ORG CHEM]  $(\text{H}_2\text{CCHOOCCH}_3)_x$  A thermoplastic polymer; insoluble in water, gasoline, oils, and fats, soluble in ketones, alcohols, benzene, esters, and chlorinated hydrocarbons; used in adhesives, films, lacquers, inks, latex paints, and paper sizes. Abbreviated PVA; PVAc. { 'pāl-i'vīn-əl 'as-ə,tāl }

**polyvinyl alcohol** [ORG CHEM] Water-soluble polymer made by hydrolysis of a polyvinyl ester (such as polyvinyl acetate); used in adhesives, as textile and paper sizes, and for emulsifying, suspending, and thickening of solutions. Abbreviated PVA. { 'pāl-i'vīn-əl 'al-kə,həl }

**polyvinyl carbazole** [ORG CHEM] Thermoplastic resin made by reaction of acetylene with carbazole; softens at  $150^\circ\text{C}$ ; has good electrical properties and heat and chemical stabilities; used as a paper-capacitor impregnant and as a substitute for electrical mica. { 'pāl-i'vīn-əl 'kär-bə,zəl }

**polyvinyl chloride** [ORG CHEM]  $(\text{H}_2\text{CCHCl})_x$  Polymer of vinyl chloride; tasteless, odorless; insoluble in most organic solvents; a member of the family of vinyl resins; used in soft flexible films for food packaging and in molded rigid products such as pipes, fibers, upholstery, and bristles. Abbreviated PVC. { 'pāl-i'vīn-əl 'klör,īd }

**polyvinyl chloride acetate** [ORG CHEM] Thermoplastic copolymer of vinyl chloride,  $\text{CH}_2\text{CHCl}$ , and vinyl acetate,  $\text{CH}_3\text{COOCH}=\text{CH}_2$ ; colorless solid with good resistance to water, concentrated acids, and alkalis; compounded with plasticizers, it yields a flexible material superior to rubber in aging properties; used for cable and wire coverings and protective garments. { 'pāl-i'vīn-əl 'klör,īd 'as-ə,tāl }

**polyvinyl dichloride** [ORG CHEM] A high-strength polymer of chlorinated polyvinyl chloride; it is self-extinguishing and has superior chemical resistance; used for pipes carrying hot, corrosive materials. Abbreviated PVDC. { 'pāl-i'vīn-əl dī'klör,īd }

**polyvinyl ether** See polyvinyl ethyl ether. { 'pāl-i'vīn-əl 'ē-thər }

**polyvinyl ethyl ether** [ORG CHEM]  $[\text{—CH}(\text{OC}_2\text{H}_5)\text{CH}_2\text{—}]_x$  A viscous gum to rubbery solid, soluble in organic solvents; used for pressure-sensitive tape. Also known as polyvinyl ether. { 'pāl-i'vīn-əl 'eth-əl 'ē-thər }

**polyvinyl fluoride** [ORG CHEM]  $(\text{—H}_2\text{CCHF—})_x$  Vinyl fluoride polymer; has superior resistance to weather, chemicals, oils, and stains, and has high strength; used for packaging (but not of food) and electrical equipment. { 'pāl-i'vīn-əl 'flūr,īd }

**polyvinyl formate resin** [ORG CHEM]  $(\text{CH}_2=\text{CHOOCH})_x$  Clear-colored resin that is hard and solvent-resistant; used to make clear, hard plastics. { 'pāl-i'vīn-əl 'fōr,māt 'rez-ən }

**polyvinylidene chloride** [ORG CHEM] Thermoplastic polymer of vinylidene chloride,  $\text{H}_2\text{C}=\text{CCl}_2$ ; white powder softening at  $185\text{--}200^\circ\text{C}$ ; used to make soft-flexible to rigid products. { 'pāl-i'vīn-il-ə,dēn 'klör,īd }

**polyvinylidene fluoride** [ORG CHEM]  $\text{H}_2\text{C}=\text{CF}_2$  Fluorocarbon polymer made from vinylidene fluoride; has good tensile and compressive strength and high impact strength; used in chemical equipment for gaskets, impellers, and other pump parts, and for drum linings and protective coatings. { 'pāl-i'vīn-il-ə,dēn 'flūr,īd }

**polyvinylidene resin** See vinylidene resin. { 'pāl-i'vīn-il-ə,dēn 'rez-ən }

**polyvinyl isobutyl ether** [ORG CHEM]  $[\text{—CH}_2\text{CHOCH}_2\text{CH}(\text{CH}_3)_2\text{—}]_x$  An odorless synthetic resin; elastomer to vis-

ous liquid depending on molecular weight; soluble in hydrocarbons, esters, ethers, and ketones, insoluble in water, adhesives, waxes, plasticizers, lubricating oils, and surface coatings. Abbreviated PVI. { 'pāl-i'vīn-əl 'ī-sə,'byūd-əl 'ē-thər }

**polyvinyl methyl ether** [ORG CHEM]  $(\text{—CH}_2\text{CHOCH}_3)_x$  A colorless, tacky liquid, soluble in organic solvents, except aliphatic hydrocarbons, and in water below  $32^\circ\text{C}$ ; used for pressure-sensitive adhesives, as a heat sensitizer for rubber, and as a pigment binder in inks and textile finishing. Abbreviated PVM. { 'pāl-i'vīn-əl 'meth-əl 'ē-thər }

**polyvinyl pyrrolidone** [ORG CHEM]  $(\text{C}_4\text{H}_5\text{NO})_x$  A white, soluble, white, resinous solid; used in pharmaceuticals, cosmetics, detergents, and foods, and as a synthetic blood plasma. Abbreviated PVP. { 'pāl-i'vīn-əl pə'räl-ə,dōn }

**polyvinyl resin** [ORG CHEM] Any resin or polymer derived from vinyl monomers. Also known as vinyl plastic. { 'pāl-i'vīn-əl 'rez-ən }

**Polyzoa** [INV ZOO] The equivalent name for Bryozoa. { 'pō-lī-zō-ə }

**polzenite** [PETR] 1. A group of lamprophyres characterized by the presence of olivine and melilite. 2. Any rock in this group. { 'pāl-zē,nīt }

**pomace** [FOOD ENG] 1. In the preparation of cider, the material derived from apples or similar fruits by crushing or grinding. 2. The residue of grape skins, seeds, and stems after grapes have been pressed and the juice has been separated. { 'pām-əs }

**Pomacentridae** [VERT ZOO] The damselfishes, a family of perciform fishes in the suborder Percoidae. { 'pō-mā-sen-trī,dē }

**Pomadasyidae** [VERT ZOO] The grunts and sweetlips, a family of perciform fishes in the suborder Percoidae. { 'pō-mā-dā-sī-ə,dē }

**Pomatiasidae** [INV ZOO] A family of land snails in the order Pectinibranchia. { 'pō-mā-tī-ās-ə,dē }

**Pomatomidae** [VERT ZOO] A monotypic family of the ciformes containing the bluefish (*Pomatomus saltatrix*). { 'pō-mā-tām-ə,dē }

**pomegranate** [BOT] *Punica granatum*. A small, deciduous ornamental tree of the order Myrtales cultivated for its fruit, which is a reddish, pomelike berry containing numerous seeds embedded in crimson pulp. { 'pām-ə,gran-ət }

**Pomeranchuk cooling** [CRYO] A method of attaining low temperatures as low as 1 millikelvin in which helium-3 is cooled by adiabatic compression at temperatures below  $0.3^\circ\text{K}$ . { 'pō-mə-rān-čuk 'kūl-īŋ }

**Pomeranchuk pole** See Pomeron. { 'pām-ə,rān-čuk,pōl }

**Pomeranchuk theorem** [PARTIC PHYS] The theorem that the total cross section both for scattering of a particle by a target particle and for scattering of its antiparticle by the target particle, approach a limit at high energies, and converge sufficiently rapidly, then these limits must be the same. { 'pō-mə-rān-čuk 'thī-rəm }

**Pomeron** [PARTIC PHYS] A Regge pole which is located at  $\alpha = 1$  in the angular momentum plane when the momentum transfer in the crossed channel equals zero, corresponding to the fact that total cross sections of reactions are observed to approach constants at high energies. Also known as Pomeranchuk pole. { 'pām-ə,rān }

**Pompe disease** [MED] A hereditary glycogen storage disease in humans arising from deficiency of a lysosomal enzyme, characterized by weakness, enlargement of the heart and liver, failure, enlargement of the tongue, and moderate enlargement of the liver. { 'pāmp di,zēz }

**Pompilidae** [INV ZOO] The spider wasps, the single family in the superfamily Pompiloidea. { 'pāmp'il-ī-ə,dē }

**Pompiloidea** [INV ZOO] A monofamilial superfamily of Hymenoptera in the suborder Apocrita with oval abdominal segments and strong spinose legs. { 'pāmp-pō'lōid-ē-ə }

**pom-pom** [ORD] 1. A rack of anti-aircraft cannon. 2. An anti-aircraft cannon. { 'pām,pām }

**PONA analysis** [ENG] American Society for Testing and Materials analysis of paraffins (P), olefins (O), naphthenes, and aromatics (A) in gasolines. { 'pō-nə-ə,'pē-ō-nə-ə-ə,səs }

**poncelet** [PHYS] A unit of power equal to the power delivered by a force of 100 kilograms-force when the point of application

# Coextrusion Developments Focus on Barrier Resins

*Improved EVOH materials, a brand-new amorphous nylon, feedblock developments and a computer program for coextruded material selection head the list of developments introduced at fall packaging conferences.*

*By Ann Brockschmidt, Executive Editor, Packaging*

The pace of coextrusion developments, while having slowed from the frantic pace of two years ago, is still active, as evidenced by the two main coextrusion conferences held this fall—SPE's Coextrusion RETEC in Arlington Heights, Ill., and the Coex '87 meeting in Teaneck, N.J., sponsored by Schotland Business Research, Inc., Princeton, N.J. Much of the news concerned materials developments, including the debut of filled EVOH resins, a new barrier amorphous nylon from Japan, and a computer program for coextrusion material selection. Machinery developments include a fixed-geometry feedblock and hints of coming developments in PVDC barrier blown films.

**New family of filled EVOH**  
Three to five times the gas barrier of other EVOH resins is claimed for a new family of EVOH grades for rigid coextruded food packaging from Du Pont, introduced at the SPE Coextrusion RETEC. Called Selar OH Plus, the new compounds are the first of a series of products resulting from proprietary blend technology that reportedly slows the permeation of gas molecules through the barrier layer.

This first series is a blend of thin, flat mica fillers in standard EVOH (Du Pont's Selar OH, with ethylene contents of 30% and 44%). The mica particles are oriented to obstruct the migration of gas molecules. The refractive index of the mica particles is 1.55, close to the EVOH index of 1.52, in order to maintain contact clarity. There are two 30% ethylene grades—3002M3 with a melt flow of 1-2 g/10 min, and 3004M3 with a melt flow of 3-4; and one 44% ethylene grade, 4408M3, with a melt flow of 8.

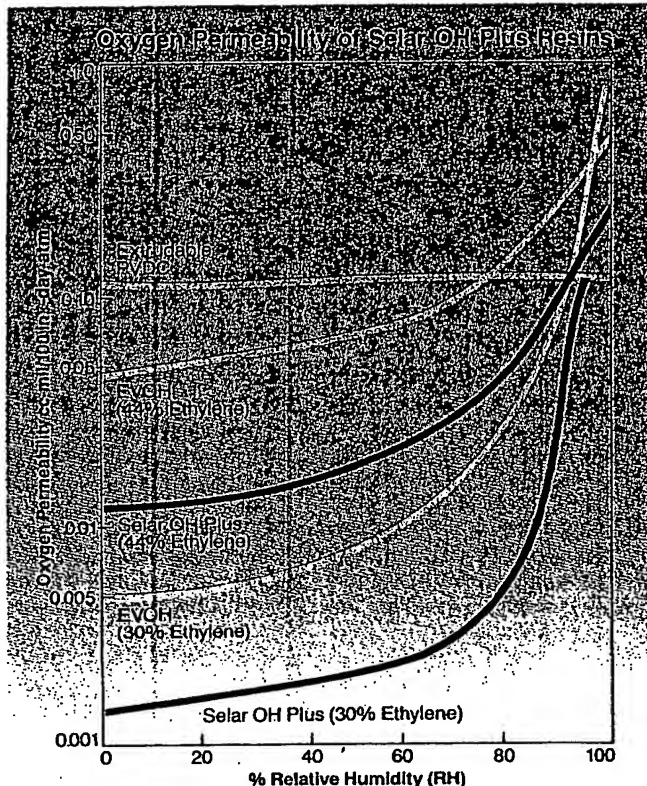


Fig. 1—New filled EVOH resins from Du Pont for rigid, coextruded food containers have three to five times the gas barrier of other EVOH resins.

Other properties of the compounds include density of 1.38, melt point of 365 F, and oxygen barrier of 0.001-0.1 cc-mil/100 in.²-day-atm over a range of 0-80% RH, as compared with 0.006-0.36 cc-mil for unfilled Selar OH (see Fig. 1). Moisture barrier is about the same as usual, though the 100%-RH curve for Selar OH is comparable to the 80%-RH curve for Selar OH Plus. Minimum thicknesses of 0.5 mils are recommended by Du Pont. Reduced flex-crack resistance restricts use of Selar OH Plus in flexible applications.

The compound processes on the same equipment as standard EVOH, though

## Barrier Packaging

the mica filler causes a slight increase in melt viscosity; for example, the Selar OH Plus grade with 30% ethylene behaves like a resin with 5% ethylene.

The new materials can either be used where extended shelf life of more than one year is required, or processors can reduce the thickness of the EVOH layer, reducing the cost of the barrier structure while retaining the gas barrier. Selar OH Plus resins sell for \$3.50/lb, vs. \$2.40/lb for Selar OH, providing twice the oxygen barrier/\$.

Still in development is an EVOH blend designed for easier processing. Thermoformers have been forced to use higher-ethylene grades (44%) for deep-draw containers. Du Pont is developing a 31%-ethylene EVOH blend that reportedly has the processibility of a higher-ethylene EVOH, with the oxygen barrier of a 30%-ethylene EVOH.

**Another new amorphous barrier nylon** An amorphous nylon that exhibits decreasing oxygen permeability with increasing humidity was introduced at Coex '87 by Mitsubishi Chemical Industries Ltd., Tokyo (U.S. offices are in White Plains, N.Y.). Novamid X21 is a copolyamide different in composition from MXD6 nylon, another barrier type that was introduced last year by Mitsubishi Gas Chemical Co. and Toyobo Co. (see *PT*, May '87, p. 17). Also, MXD6 is semicrystalline and therefore can't be transparent in high humidity without further post-processing such as orientation.

Mechanical properties of X21 are almost the same as those of nylon 6, but its rate of water absorption is much

## Coextrusion Trend to Simpler Structures?

Two-layer, scrapless and recyclable are the key words in the future of food packaging, according to GE's R. P. Hop, manager of Gepax programs, GE Plastics, Pittsfield, Mass. In a recent interview with *PE*, he discussed how he sees the future of multilayer packaging and where GE is putting its packaging R&D efforts.

The number-one priority at Gepax is development of a scrapless process capable of producing 130-150 million multilayer containers/yr for food and beverage packaging. To this end, GE invited four machine builders to bid on the project and has now settled on two of them, one in the U.S. and the other in Europe. GE has acquired worldwide patents and all existing equipment for a scrapless, "gateless" process that has so far been developed for low-volume outputs. Hop wouldn't say more about the process, except that it is unlike any conventional plastics manufacturing process. The machine builders, along with two end users in different markets, are currently working to increase the output of this process. GE has made single-layer containers on a scrapless machine, filled and successfully tested them, says Hop.

Hop believes in multilayer structures for the long run—two-layer structures, that is. He doesn't think it makes sense to try to develop one resin that can do it all—provide gas and moisture barrier, heat resistance, stiffness, impact strength, etc., though there may be some applications where that is practical. The ultimate structure for the majority of applications will be two layers—a thin barrier layer and another for structural purposes. GE is focusing its efforts on development of improved barrier resins, including modifications of polycarbonate and thermoplastic polyester that do not require tie layers, for economics and also for ease of recycling. Compatibility with recycle is also a key requirement of GE's barrier-resin development program.

For the short term, GE is working to simplify current multilayer structures. The company is not involved with any seven-layer structures and is working to remove one layer in current five-layer structures. "Five-layer structures are painful from a financial standpoint in terms of equipment cost," states Hop. "We will be ready to talk about developments in this area next year."

Hop described barrier container development over the long term as a progressive reduction of layers. Present barrier containers have three, five or seven layers. Now in development is a barrier polycarbonate for multilayer structures with fewer components. After that will come the type of two-layer structures mentioned above, employing an as-yet-to-be-announced barrier resin. The final evolutionary step will be development of a super barrier resin that will be able to stand alone, in some applications.

Much has been written about GE's plan to recycle automotive parts. Hop outlined GE's plans for recycling packaging made of its engineering resins. The company will buy back consumer recycle (f.o.b. Pittsfield) and has developed proprietary recycle technology over the last 1½ years that can produce "practically virgin" material—meaning at most a 5% loss of properties. GE has built a small pilot plant for polycarbonate to evaluate the technology. Besides being reused in its original applications, the recycled material may be used in composite materials such as Azdel (a product of the GE/PPG joint venture), building and construction applications, and automotive.

Recycled polycarbonate can command a buy-back price of 70-90¢/lb, according to Hop, compared with \$2/lb for virgin. That's a much better value than what's possible with PET recycle. PET bottle resin costs 60-70¢/lb for virgin. The compounding cost alone is 15¢/lb, not including cleaning, base cups, etc., according to Hop, and repelletized resin is selling for 25-26¢/lb.

lower; X21 absorbs about 1.5% by weight under the same conditions that nylon absorbs 3%.

As shown in Fig. 2, oxygen permeability of X21 is nearly the same as that of EVOH or MXD6 nylon under high humidity, while under normal humidity (65-75% RH), it becomes intermediate between oriented PET and oriented nylon on the one hand, and MXD6 and EVOH on the other. It's very unusual for the oxygen permeability of nylon to decrease with increasing humidity. Mitsubishi speculates that this is because the resin has a slight tendency to crystallize. The transition from amorphous to semi-ordered state might occur with mild water absorption, and the barrier improvement might be derived from this roughly ordered state.

Novamid X21 is mostly compatible with nylon 6 and is capable of reducing the latter's thermoforming temperatures and increasing the shrinkage of

oriented film, as well as improving transparency and barrier properties. In tests on monolayer injection stretch-blow molded bottles, oxygen permeability of an X21 bottle was half that of a

PET bottle. The X21 bottle also demonstrated superior chemical resistance and heat resistance (the bottle was not deformed by filling with 194 F water).

Mitsubishi developed Novamid X21 in 1985 and now has a pilot plant in Japan; sample quantities are available in the U.S. It is priced at \$4/lb. Novamid X21 is currently being used in Japan for coextruded film packaging for processed meat.

**Polycarbonate for PVDC coextrusion**  
Mobay Corp. discussed the use of its new Makrolon DP-1-1073 polycarbonate resin, developed for coextrusion with PVDC, at the Coextrusion RE-TEC. As previously reported (*PT*, July '87, p. 12), the new polycarbonate can be extruded at temperatures as low as 415 F. Some of its physical properties are slightly different from normal polycarbonate—e.g., impact strength is 14 ft-lb/in., vs. 16 ft-lb/in. for typical PC.

Yellowness Index of Polycarbonate/PVDC Coextrusion<sup>a</sup>

|  | Pre Retort | Post Retort <sup>b</sup> |
|--|------------|--------------------------|
| 36 mil<br>Original (as received)                               | 36.31      | 64.20                    |
| 36 mil<br>Dried 250 F, 45 min                                  | 43.39      | 70.10                    |
| 34 mil<br>Dried 250 F, 45 min<br>Thermoformed 446 F,<br>24 sec | 34.54      | 62.12                    |
| 17.5 mil<br>Thermoformed Part                                  | 34.29      | —                        |

<sup>a</sup>Mobay Corp.

<sup>b</sup>Retort conditions: 250 F, 45 min, 19 psi



Mobay tested rigid PC/PVDC structures with several tie materials and all exhibited excellent adhesion to both resins, though Mobay declines to identify them. A tie layer may not be needed at all, according to Mobay.

The PC/PVDC coextrusion does become yellower when retorted (see table), but Mobay's evaluations show no significant loss of barrier. As for thermoforming, no PVDC degradation in a 36-mil PC/PVDC coextrusion was observed under typical polycarbonate thermoforming conditions, and materi-

neck area to obtain the heat resistance and dimensional stability required for sealability. Hoechst-Celanese makes a polyarylate called Durel.

**Computer program for coextruded materials** Du Pont has developed an interactive computer program to aid in materials selection for coextruded barrier plastic containers. The artificial intelligence system, called Packaging Advisor, helps users determine the most economical package structure to meet their gas-barrier requirements.

## Barrier Packaging

can be updated as new developments occur.

**Fixed-geometry feedblock** Cloeren Co. announced at Coex '87 that it's now offering a fixed-geometry feedblock for large-volume applications such as aseptic packaging, to eliminate operator error. It is a "hybrid" design, stemming from the company's vane technology. Inside the blocks, "die lips" are located at the point of convergence where two flow channels come together. The die lips are externally accessible, so that they can be replaced. In high-performance applications, the cost is comparable to that of the vane feedblock; for custom sheet applications, cost could be up to 20% less.

**PVDC blown film developments** Now that Dow Chemical has commercialized pelletized forms of its standard Saran PVDC and of a new higher-barrier methyl acrylate PVDC copolymer, extrusion equipment suppliers may be developing new designs to take advantage of the pellets' improved processability. At least one firm, which did not want to be identified, sees a need to develop blown film equipment to handle the "new" PVDC. According to this source, standard blown film lines can't run PVDC, even in pellet form, because residence times are too high. In order to reduce the residence time from the usual 6-10 min down to 2 min, this firm believes the answer is a shallow, short screw with 18-24:1 L/D, and very short coextrusion adapters and die channels. Also under consideration by this firm is a tubular biaxial orientation process to achieve higher machinability (stiffness) in coextruded films containing PVDC.

There's evidence that another major Dow R&D program—on PVDC recycling—has achieved commercial fruition. Ball Corp.'s Plastics Div., Evansville, Ind., has a PVDC recycling program that has successfully achieved commercial output rates—in excess of 1000 lb/hr—of a PP/PVDC structure containing recycle. A Ball source says the company is currently working with customers to develop commercial applications. □□

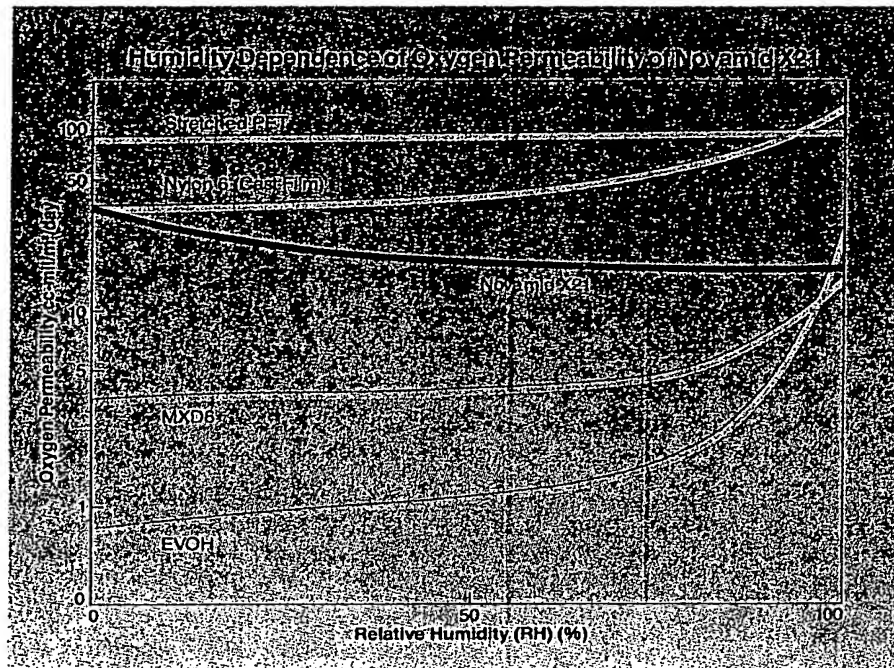


Fig. 2—A new amorphous barrier nylon, Novamid X21, exhibits the unusual characteristic of decreasing oxygen permeability with increasing humidity.

al distribution appears to have been uniform and indicates a good flow of all layers. Mobay evaluated a thin, shallow tray, cup step mold, deep-dish bowl, and a shape that resembles a bell.

Mobay is now evaluating long-term production feasibility of PC/PVDC coextrusions and use of regrind.

**Polyarylate coextrusion yields heat-resistant bottle** The Engineered Plastics Div. of Hoechst-Celanese Corp. reported at Coex '87 another sign that engineering thermoplastics are finding a niche in disposable packaging for high-heat applications. Yamamura Glass Co. of Japan has developed a five-layer bottle of PET/polyarylate/PET that will withstand hot filling to 195 F while maintaining its clarity. Additionally, unlike conventional heat-resistant PET bottles, no post-mold heat-treatment is required to crystallize the bottle

The system will be licensed to Du Pont customers. It performs barrier-vs.-cost calculations and tabulates the data in a way that allows the user to consider all the available alternatives. It considers the container fabrication process, filling/sterilization process, recycle level, package storage condition, need for tie layers and need for optical properties. Users can customize the program with their own cost and performance data. This program is a companion to the "Smart Choice" program designed to aid selection from Du Pont's nine families of Bynel tie resins. Both programs

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Mitsubishi Chemical Industries, White Plains, N.Y. (CIRCLE 38)  
Mobay Corp., Pittsburgh (CIRCLE 39)

Ref. C

# United States Patent [19]

[11]

4,252,907

Ogasa

[45]

Feb. 24, 1981

## [54] PROCESS FOR PREPARING A POROUS COMPOSITE MATERIAL

[75] Inventor: Tatsuo Ogasa, Yokohama, Japan

[73] Assignee: Director General of Agency of Industrial Science and Technology, Tokyo, Japan

[21] Appl. No.: 72,185

[22] Filed: Sep. 4, 1979

### [30] Foreign Application Priority Data

Nov. 18, 1978 [JP] Japan ..... 53-142522

[51] Int. Cl.<sup>3</sup> ..... C08J 9/08; C08J 9/10

[52] U.S. Cl. .... 521/92; 521/65; 521/72; 521/134; 521/139; 521/140; 521/905

[58] Field of Search ..... 521/72, 65, 92, 134, 521/905, 139, 140

### [56] References Cited

#### U.S. PATENT DOCUMENTS

3,027,336 3/1962 Götz ..... 521/905

|           |        |                |         |
|-----------|--------|----------------|---------|
| 3,795,720 | 3/1974 | Schwartz ..... | 521/134 |
| 3,816,170 | 6/1974 | Mudde .....    | 521/134 |
| 3,873,476 | 3/1975 | Jabs .....     | 521/905 |
| 3,879,493 | 4/1975 | Mudde .....    | 521/905 |
| 3,888,766 | 6/1975 | De Young ..... | 521/905 |

Primary Examiner—Morton Foelak

### [57] ABSTRACT

A porous composite material includes a porous body formed of a hydrophobic polymer such as polyvinyl chloride and a layer of a hydrophilic polymer such as polyvinyl alcohol provided over at least a portion of the interior surface of each of the pores of the porous body. The composite material is obtained by a process including the steps of dispersing an aqueous solution containing a hydrophilic polymer and a foaming agent into a solution of a hydrophobic polymer in an organic solvent to form an emulsion, removing the water and the organic solvent from the emulsion to obtain a solid, and subjecting the solid to foaming conditions.

7 Claims, No Drawings

the solid mass according to the kind of the foaming agent employed. The conditions for and the manner of the foaming treatment are well known in the art.

Thus, a multiplicity of pores are formed within the hydrophobic polymer body. Each pore is in communication with the surface of the body through a path formed by the gases generated and escaped to the air during the treatment of the foaming agent. Further, since these pores are produced with the portions corresponding to the liquid particles containing the hydrophilic polymer and the foaming agent as cores, at least a part of the interior surface of each pore is covered with a layer of the hydrophilic polymer. As a result of this structure, the porous composite material produced in accordance with the process of the present invention has an improved hygroscopic property inherent to the hydrophilic polymer exposed to the surfaces of the pores.

The porous composite material of this invention may be applied to a variety of fields. For example, films or sheets of the composite material can be used as interior decoration materials which also serve to prevent dew from being formed thereon. The porous composite material can be provided over the surface of a substrate to form a composite article. Such article may be advantageously produced by applying a coating of the above-described emulsion onto the substrate, drying the coating and subjecting the dried coating to a foaming treatment.

The following examples will further illustrate the present invention.

#### EXAMPLE 1

100 parts by volume of a solution of polyvinyl chloride in ethylene dichloride (concentration of the polymer in the solution was 100 g/l), 10 parts by volume of an aqueous solution of polyvinyl alcohol (concentration: 20 g/l) and 15 parts by volume of an aqueous potassium bicarbonate solution (concentration: 250 g/l) were mixed together for emulsification. The emulsion was applied onto a glass plate, which was then placed in a freezer at  $-20^{\circ}\text{C}$ . for 48 hours. Thereafter, the emulsion was dried at  $60^{\circ}\text{C}$ . under vacuum to obtain a film of 0.5 mm thick. The film, after being separated from the glass plate, was then immersed in 2 N HCl for 48 hours to allow to generate  $\text{CO}_2$  by decomposition of the potassium bicarbonate. After washing with water, the resulting film was dried under vacuum to obtain a porous composite film having a density of 0.58. The porous composite film had a porosity of 60% and the diameter of the pores was found to range from 5 to  $50\mu$ . When the composite film was kept at  $30^{\circ}\text{C}$ . and under a constant relative humidity of 85% and 91%, the film absorbed moisture in an amount of 21% based on its dry weight in the case of 85% humidity and 25% in the case of 91% humidity.

#### COMPARATIVE EXAMPLE

For the purpose of comparison, a porous film was prepared in the same manner as in Example 1 except that no polyvinyl alcohol was employed. The film had a porosity of 30%. A moisture absorb test revealed that the film absorbed moisture in an amount of 9% based on its dry weight when the film was allowed to stand under conditions of  $30^{\circ}\text{C}$ . and 91% humidity.

#### EXAMPLE 2

Porous composite films were produced by the same way as in Example 1 except that the weight ratio of  $\text{KHCO}_3/\text{PVC}$  was varied. The porosity of the film in the case of  $\text{KHCO}_3/\text{PVC}$  ratio of 0.63 was 69% and in the case of  $\text{KHCO}_3/\text{PVC}$  ratio of 0.19 was 45%.

#### EXAMPLE 3

Example 1 was repeated using polyacrylic acid and polyvinyl pyrrolidone, respectively, in place of the polyvinyl alcohol. The amounts of moisture absorbed in the resulting porous composite films, when placed under conditions of  $30^{\circ}\text{C}$ ., 91% relative humidity, were 21% in the case of polyacrylic acid and 22% in the case of polyvinyl pyrrolidone, based on the dry weight of respective films.

What is claimed is:

1. A process for the production of a porous composite material, comprising the steps of:

emulsifying an aqueous solution containing a hydrophilic polymer and a foaming agent into a solution of a hydrophobic polymer in an organic solvent to form an emulsion in which liquid particles of said aqueous solution are dispersed in said organic solution;

removing said organic solvent and water from said emulsion to obtain a solid; and  
subjecting said solid to foaming conditions thereby to obtain the porous composite material.

2. A process as set forth in claim 1, wherein said emulsion forming step includes mixing the aqueous solution containing 1 to 20 wt % of the hydrophilic polymer and at least 5 wt % but up to the saturated concentration of the foaming agent with the organic solution containing 1 to 30 wt % of the hydrophobic polymer with a mixing ratio by weight of the former solution to the latter solution in the range of 2 to 20:100.

3. A process as set forth in claim 1, wherein the hydrophilic polymer is used in an amount of 1 to 25 parts by weight per 100 parts by weight of the hydrophobic polymer and the foaming agent is used in an amount of 2 to 70 parts by weight per 100 parts by weight of the hydrophobic polymer.

4. A process as set forth in claim 1, wherein the hydrophobic polymer is a resin selected from the group consisting of polyolefin resins, styrene resins, acrylic resins and polyvinyl chloride resins.

5. A process as set forth in claim 4, wherein the hydrophobic polymer is a member selected from the group consisting of polyethylene, polypropylene, polybutene, ethylene-propylene copolymers, polystyrene, acrylonitrile-butadiene-styrene copolymers, polyacrylonitrile, a polyacrylic acid ester, polyvinyl chloride and polyvinylidene chloride.

6. A process as set forth in claim 1, wherein the hydrophilic polymer is a member selected from the group consisting of polyvinyl alcohol, polyacrylic acid, polyvinyl pyrrolidone, polyethylene glycol and a salt of polyacrylic acid.

7. A process as set forth in claim 1, wherein the foaming agent is a member selected from the group consisting of potassium bicarbonate, sodium bicarbonate, azodicarboamide, diphenylsulfone-3,3'-disulfohydrazine and benzenesulfonic acid diphenylhydrazine.

\* \* \* \* \*

# United States Patent [19]

Rechlicz et al.

[11]

4,065,534

[45]

Dec. 27, 1977

## [54] METHOD OF PROVIDING A RESIN REINFORCED ASBESTOS DIAPHRAGM

[75] Inventors: Thomas A. Rechlicz; Bernard A. Maloney, both of Corpus Christi, Tex.

[73] Assignee: PPG Industries, Inc., Pittsburgh, Pa.

[21] Appl. No.: 678,561

[22] Filed: Apr. 20, 1976

[51] Int. Cl.<sup>2</sup> ..... C25B 13/06

[52] U.S. Cl. .... 264/91; 162/155; 204/296; 264/104; 264/115; 264/127; 264/136; 264/257; 264/331

[58] Field of Search ..... 264/104, 103, 109, 115, 264/125-126, 127, 232, 233, 331, 136-137, 257, 91; 162/3, 153, 155; 204/296

[56]

## References Cited

### U.S. PATENT DOCUMENTS

|           |         |                        |         |
|-----------|---------|------------------------|---------|
| 3,694,281 | 9/1972  | Leduc .....            | 204/296 |
| 3,723,264 | 3/1973  | Leduc et al. ....      | 204/295 |
| 3,853,721 | 12/1974 | Darlington et al. .... | 204/296 |
| 3,945,910 | 3/1976  | De Ceuster et al. .... | 204/296 |
| 3,980,613 | 9/1976  | Bachot et al. ....     | 264/127 |

### FOREIGN PATENT DOCUMENTS

2,401,942 8/1974 Germany.

Primary Examiner—Jeffery R. Thurlow

Attorney, Agent, or Firm—Richard M. Goldman

[57]

## ABSTRACT

Disclosed is a method of preparing an asbestos diaphragm containing a thermoplastic resin. According to the method disclosed herein, an asbestos mat is prepared by drawing the asbestos mat from a salt free, aqueous, asbestos slurry, providing the resin, and thereafter heating the mat to form the resin reinforced mat.

5 Claims, No Drawings



and electrolysis was commenced. After 88 days of electrolysis at 190 amperes per square foot, the average cell voltage was 3.19 volts, the average cathode current efficiency was 97.31 percent, and the cathode kilowatt hours per unit of  $\text{Cl}_2$  and  $2\text{NaOH}$  was 2245 kilowatt hours per unit.

### EXAMPLE III

Codeposited asbestos-resin diaphragms were drawn from salt-free sodium hydroxide slurries and tested as diaphragms in laboratory chloralkali cells.

Slurries were prepared by adding Quebec Asbestos Producers Association grade 4D-12 chrysotile asbestos to 10 weight percent solutions of sodium hydroxide. Thereafter, a dispersion of Allied Chemical Corporation HALAR (TM) copolymer of ethylene and chlorotrifluoroethylene with Triton X-100 surfactant in water was added to the slurry in sufficient quantity to provide a solids content (basis total weight of asbestos and resin) that was 10 weight percent resin and 90 weight percent asbestos. The slurry was agitated and then deposited on cathodes as described in Example I above. The resulting codeposited asbestos and resin mats were then heated to the temperatures and for the times indicated in Table I below.

The cathode units were assembled with anode units as described in Example I above and electrolysis was commenced at 190 amperes per square foot. The results shown in Table I were obtained.

TABLE I

| Run                          | A       | B       |
|------------------------------|---------|---------|
| Time of heating (hours)      | 5.8     | 5.8     |
| Temperature (degrees C.)     | 250-260 | 250-260 |
| Days on line                 | 194     | 196     |
| Average Cell Voltage (volts) | 3.13    | 3.09    |
| Cathode efficiency (%)       | 96.13   | 95.33   |
| Kilowatt hours/unit          | 2228    | 2226    |

### EXAMPLE IV

A codeposited asbestos-resin diaphragm was drawn from a salt-free sodium hydroxide slurry and tested as a diaphragm in a laboratory chloralkali cell.

The slurry was prepared by adding Quebec Asbestos Producers Association grade 4D-12 chrysotile asbestos to a 10 weight percent solution of sodium hydroxide. Thereafter, a dispersion of Allied Chemical Corporation HALAR (TM), a copolymer of ethylene and chlorotrifluoroethylene, with duPont MERPOL (TM) SE surfactant in water was added to the slurry in sufficient quantity to provide a solids content (basis total weight of asbestos and resin) that was 10 weight percent resin and 90 weight percent asbestos. The slurry was agitated and then deposited on a cathode as described in Example I above. The resulting codeposited asbestos and resin mat was then heated to the temperature and for the time indicated in Table II below.

Thereafter a solution prepared from 5.1 grams of Allied Chemical Corporation KYNAR (TM) RC 9332 polyvinylidene fluoride in 200 milliliters of 5 weight percent sodium hydroxide was poured over the cathode and a vacuum was drawn within the cathode to pull the solution through. Thereafter, the cathode was heated. The vacuum, the time of heating, and the temperature are shown in Table II below. The cathode and an anode were then assembled to form a cell as described in Example I above. The results obtained are shown in Table II below.

TABLE II

Time of first heating (hours)

1

TABLE II-continued

|                                   |       |
|-----------------------------------|-------|
| Temp. of first heating (° C.)     | 263   |
| Vacuum (cm Hg)                    | 25    |
| Time of second deposition (hours) | 0.33  |
| Temp. of second heating (° C.)    | 200   |
| Days on line                      | 97    |
| Average cell voltage (volts)      | 3.20  |
| Cathode efficiency (%)            | 97.36 |
| Kilowatt hours/unit               | 2251  |

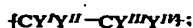
Although the method of this invention has been described and illustrated with respect to particular exemplifications and embodiments thereof, it is not intended to be so limited but is to only be limited as described in the appended claims.

We claim:

1. In a method of preparing an asbestos diaphragm containing a thermoplastic (hydrophobic resin) which method comprises forming a fibrous chrysotile asbestos mat having the resin therein and thereafter heating the mat to melt said resin, the improvement wherein said resin is capable of being oxidized and degraded when heated above the crystalline melting point thereof in the presence of asbestos, and which method comprises depositing said asbestos diaphragm from an aqueous slurry substantially free of alkali metal chloride and consisting essentially of from about 1 to about 30 weight alkali metal hydroxide and from about 0.1 to about 10 weight percent total asbestos and resin, wherein the resin is from about 0.2 to about 8 weight percent of the total asbestos and resin and wherein the resin is selected from the group consisting of:

A. hydrocarbon resins;

B. homopolymers having the empirical formula:



and

C. copolymers having hydrocarbon and halocarbon moieties wherein the halocarbon moiety is chosen from the group consisting of halocarbons having the empirical formula:



wherein at least 20 percent of the copolymer is the hydrocarbon moiety;

where  $Y^I$  is halogen chosen from the group consisting of fluorine, chlorine, and bromine,  $Y^II$ ,  $Y^III$ , and  $Y^IV$  are chosen from the group consisting of fluorine, chlorine, bromine, and hydrogen, and at least one of said  $Y^II$ ,  $Y^III$ , and  $Y^IV$  is hydrogen, and where  $X^I$  is a halogen chosen from the group consisting of fluorine, chlorine, and bromine, and  $X^II$ ,  $X^III$ , and  $X^IV$  are chosen from the group consisting of fluorine, chlorine, bromine, and hydrogen.

2. The method of claim 1 wherein the concentration of alkali metal chloride in the slurry is low enough to avoid crystallization of solid alkali metal chloride on the asbestos fibers.

3. The method of claim 1 wherein the alkali metal hydroxide is sodium hydroxide.

4. The method of claim 1 wherein the resin is a homopolymer chosen from the group consisting of polyvinyl chloride, polyvinylidene chloride, polytrichloroethylene, poly(1-chloro-2,2-difluoroethylene), poly(1-chloro-1,2-difluoroethylene), polytrifluoroethylene, polyvinyl fluoride, poly(vinylidene fluoride), polyethylene, polypropylene, polyisobutylene, and polystyrene.

5. The method of claim 1 wherein the hydrophobic resin is a copolymer of a hydrocarbon and a halocarbon selected from the group consisting of perfluoroethylene, trifluoroethylene, vinylidene fluoride, vinylidene chloride, and chlorotrifluoroethylene.

\* \* \* \* \*